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October 1, 1980

BRIEFING MEMO ON INDUSTRIAL PROCESS USED AT REILLY

TAR & CHEMICAL CORPORATIONS AT ST. LOUIS PARK, MINNESOTA PLANT

(1916-1973)

This memo will present the information available regarding the industrial processes carried out at the Reilly Tar and Chemical Corporation's St. Louis Park plant during its approximately 57 years of operation. Information regarding raw materials, refinery processes and products, creosoting plant (Republic Creosoting Company) processes and products, and wastewater treatment processes will be presented. From the information available to us, the known changes, disruptions and modifications to these processes will be traced through the history of the plant. In some cases, assumptions as to some of the minor processing steps will be made in an attempt to present a clear and complete picture of the plant operations.

For the purposes of clarity, this memo is divided into three main sections: 1) coal tar refinery operations, 2) creosoting plant operations, and 3) wastewater treatment plant operations. Each of these subjects will be presented with attention given to raw materials, processes, products, historical events, such as accidents, explosions, and process modifications, and state-of-the-art technology available to minimize pollution potential.

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1) Coal Tar Refinery

a) Raw Materials

The major raw materials used in the refining process at the St. Louis Park plant were various types of coal tar, and water gas tar (which in the literature is something deemed to be different from coal tar as it also contains some petroleum products) and oil gas tar. Other raw materials include pitch, caustic soda and sulfuric acid, and fuel oil for heating the stills. In the mid to late 1950's fuel oil was replaced by natural gas which apparently was used until the closure of the plant except during some of the winter months when the gas supply was unavailable. At these times fuel oil was again used.

The coal tars used varied throughout the years of plant operations. Sources were numerous and apparently chosen on the basis of cost from month to month during the earlier years of operation. The sources became more consistent in the mid-50's as did the products of the plant. From 1953 until plant closure, coke oven tar was almost exclusively the coal tar used.

The types of coal tar used at the plant were coke oven tar, vertical retort tar and water gas tar. At times on the weekly reports, the tar is merely referred to as coal tar. It appears that usually this is coke oven tar, although other

forms may have been included to a small extent. The following paragraphs describe the sources of the various types of tars and the important characteristics of each.

Before discussing the various types of coal tar, I will present some of the typical characteristics and composition of coal tar. The following chart has been taken from "Asphalts and Allied Substances" by Herbert Abraham, Sixth Edition, Copyright 1961. As you will note many of the components are listed as "unidentified". As of 1966 only about 215 compounds from coal tar had been identified with researchers estimating that approximately 5000 compounds comprised coal tar.

The chart which follows was modified by adding typical temperature ranges at which the fractions are collected in the coal tar distillation process. The compounds presented as phenols, phenol homologues, tar bases, yellow solids of pitch oils, pitch greases, and resinous bodies can be found in varying amounts in more than one of the major fractions (light oil, middle oil, heavy oil, anthracene oil, and pitch).

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Percent:

Light oil (up to 200°C):

Crude benzene and toluol	0.3
Coumarone, indene, etc.	0.6
Xylenes, cumenes and isomers	1.1

Middle (200°- 250°C) and heavy (250°-300°C):

Naphthlene	10.9
Unidentified oils in range of naphthalene and methylnaphthalenes	1.7
a-Monomethylnaphthalene	1.0
b-Monomethylnaphthalene	1.5
Dimethylnaphthalenes	3.4
Acenaphthene	1.4
Unidentified oils in range of acenaphthene	1.0
Fluorene	1.6
Unidentified oils in range of fluorene	1.2

Anthracene oil (300°-350°C or end of distillation):

Phenanthrene	4.0
Anthracene	1.1
Carbazol and kindred nonbasic nitrogen- containing bodies	2.3
Unidentified oils in range of anthracene	5.4

<u>Phenol</u>	0.7
<u>Phenol homologues</u> (largely cresols and xylenols)	1.5
<u>Tar bases</u> (mostly pyridine, picolines, lutidines, quinolines and acridine)	2.3
<u>Yellow solids of pitch oils</u>	0.6
<u>Pitch greases</u>	6.4
<u>Resinous bodies</u>	5.3
<u>Pitch</u> (460° F. fusing-point)	44.7

TOTAL 100.00

(See Appendix A for chemical structure of the major compounds in this table.)

Coke oven tar is that portion of bituminous coal burned in a coke oven which volatilizes (or vaporizes) upon heating at temperatures between 1000 and 1300°C and which is condensed as the gases are cooled. Coke oven tar is typically produced at the

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highest temperatures of the types of coal tar; since certain chemical reactions occurring in the coal are dependent on temperature, this high temperature greatly influence the composition of the coke oven tar. Since temperatures exceeding 600°C favor the formation of aromatic compounds (those containing one or more benzene rings), most of the compounds present in coke oven tar are aromatic. The higher temperatures in the coking process as compared to other coal tar-producing processes also lead to increased formation of aromatic compounds containing two or more benzene rings. Thus naphthalene (a two-ring compound) is much more prevalent in coke oven tar than in other types of coal tar. Higher weight aromatics should also be more prevalent in coke oven tar. These include anthracene and phenanthrene (3-ringed compounds) and their derivatives and naphthcene, chrysene, triphenylene, and pyrene (4-ringed compounds) and their derivatives. The high temperature in a coking oven also leads to lower levels of tar acids and bases than in other tars.

Vertical retort tar was produced by the illuminating gas manufacturing industry. The gases given off by bituminous coal when heated are collected and the tar is removed in various cooling devices. Since the gas is the principal product different conditions are necessary in the retort in which the coal is heated. Vertical retorts usually heat the coal to temperatures of 1000°-1200°C. Typically vertical retort tar has a lower specific gravity than does coke oven tar (1.08-1.16

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versus 1.15-1.26). Naphthalene is almost nonexistent and tar acids are significantly higher than in coke oven tar. (Ref. 2, page 77). Data produced in the Republic Creosoting Company Company's laboratory on January 6, 1942 (Document #214588) presents the specific gravity of vertical retort tar used at the St. Louis Park plant as 1.086. This document presents little other information which would be useful to our purposes.

Water-gas tar is produced by the heating of coke or anthracite coal and passing steam and air over the surface. Also, petroleum products are used in the gas production process. Temperatures in the production apparatus are maintained at approximately 640°-700°C. For a more detailed description of the water-gas production process, see Ref. 2, pp. 102-104. ✓

Water-gas tars are lighter than other tars. Significant compounds present in the tar are benzene, toluene, xylenes, naphthalene, and anthracene. The tars include negligible amounts of phenols and bases.

The relative use of the three main types of tars used at St. Louis Park is presented on Figure 1 along with total tar use. This figure was prepared using data from Document #400500 entitled "St. Louis Park Plant Tar Purchases: presumably prepared by the Company. This data only covers the time period of 1934-1951. Oil-gas tars were also used during the 1920's when

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coal tar was unavailable.

Data was also gathered from the weekly production reports on the refinery that we have. This data, while not complete, was used to determine trends in tar usage at the plant. The data is available from reports from 1934-1970. It agrees quite well with the data presented in Figure 1 for the years 1934-1951. Therefore it seems likely that we can draw some general conclusions from it.

From 1934 until the early 50's (and probably before this period) coke oven tar comprised about 60-75% of the total tar refined at the plant. During this period the amounts of water-gas tar and vertical retort tar varied significantly but both averaged about 10-20% of total tar used. The years 1940-1945 were peak production years for the refinery probably due to the war. It seems that the Company bought tar up until the 50's mainly on the basis of cost due to the large number of suppliers and the diversity in types of tar used.

From the early 50's to about 1960, about 90-95% of the tar used appears to have been coke oven tar. After 1960, there is no more mention of the other tars in the reports that I have seen, so I will assume that the Company had become pickier about the tar characteristics and uniformity. During the 60's only two major tar sources are mentioned (Duluth and Steelton). It seems that the products produced at St. Louis Park also became less variable during this time.

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Refining levels fell during the late 40's and early 50's from the peak years of the early 40's, during which approximately 100,000 gallons of tar per week was processed. The lower level was about 50,000 gallons per week. The late 50's and early 60's showed average rates of refining of about 90,000 gallons per week with peak years up around 120,000 gallons per week. The mid 60's and early 70's (until plant closure) were the years of maximum production with refining rates averaging 120,000 - 130,000 gallons per week.

b) STORAGE OF RAW MATERIALS

The following document presents the storage facilities at the plant as of February 24, 1936. At that time the tanks listed as Sty. 1-10 and the cistern were presumably used to store the various types of raw tars used at the plant. Also, some of the major products such as creosote oil must have been stored in some of these tanks.

The tar cistern was a steam heated, in-ground covered storage facility. The composition of the sides and bottom of the cistern is unknown.

The second document entitled "Tank Data - St. Louis Park", dated January 31, 1961, shows which tanks were used for which raw tars, fuels and products. We can deduce that most of these tanks were at least 35 years old at the time. A 35 year old steel tank (and many were probably about 53 years old-the refinery's age) is much more apt to be a "leaker" than a new tank.

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**ST. LOUIS PARK PLANT
Capacity of Tanks**

February 21, 1936
(See also 2-12-36 letter)

**Capacity of Storage Tanks at St. Louis Park Refinery
and By-Products**

Cistern	223,722 gals.	Underground Tanks at Refinery
Stg. 1	47,040	1 3,120 gals.
Stg. 2	100,920	2 2,880
Stg. 3	533,129	3 13,070
Stg. 4	253,490	4 14,610
Stg. 5	252,183	5 5,200
Stg. 6	252,185	6 14,420
Stg. 7	101,730	7 10,716
Stg. 8	101,730	8 10,716
Stg. 9	50,589	9 Not in
Stg. 10	50,589	10 Not in
		11 Not in
		12 Not in
Funnel Tanks at By-Products		13 4,260
1	3,274 gals.	14 Not in
2	3,274	15 Not in
3	1,730	16 12,110
4	1,422	17 5,170
		18 5,170
Other By-Products Tanks		
A	10,010 gals.	Underground Tanks at By-Products
B	10,010	FFPO 7,840 gals.
C	6,276	J 13,490
D	5,128	
E	9,982	
G	11,200	
H	8,084	
K	5,810	
I (H ₂ SO ₄)	8,400	

**CRYSTALIZING PLANT
Storage Tanks**

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#1 Treating Tank	46,998 gals.
#2 "	35,249
#3 "	47,785
#4 "	40,998
#5 "	46,028
Fuel Oil Ground Tank	1,391
ZnCl ₂ Ground Tank #1	10,152
" " #2	13,152

See Main Office permanent file "Storage Tank Capacity".

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REFINERY - OUTSIDE TANKS

January 31, 1961

NO.	PRESENT USE	DIAMETER	HEIGHT OR LENGTH	CAPACITY (Gallons)
1	Water Gas Tar	20' 0"	20' 0"	47,040
2	Creosote Oil	23' 11"	28' 6"	95,788
3	Creosote Oil	54' 1"	30' 0"	539,930
4	Coke Oven Tar	38' 0"	29' 0"	246,036
5	Coke Oven Tar	40' 0"	25' 0"	235,000
6	Coke Oven Tar	40' 0"	25' 0"	235,000
7	Coke Oven Tar	27' 4"	23' 0"	100,924
8	Creosote Oil	27' 4"	23' 0"	100,924
9	Creosote Oil	21' 1"	20' 0"	50,580
10	Creosote Oil	21' 1"	20' 0"	50,580
11	Road Tar Flux	7' 8"	28' 2"	10,047
12	Creosote Oil	7' 8"	28' 2"	10,047
13	Creosote Oil	7' 2"	32' 3"	10,069
14	Creosote Oil	7' 5"	32' 2"	10,624
15	Creosote Oil	7' 8"	28' 2"	10,032
16	Road Tar	7' 8"	28' 2"	10,032
Cistern	Coke Oven Tar	100' 1" x 58' 8" x 7' 7"		333,000
18	Roofing Pitch	12' 6"	28'	25,704
19	Gary Pitch	12' 6"	28'	25,704
20	Electrode Binder Mix	15' 0"	23'	30,404
RCX-19	Gasoline	93"	33 1/2'	12,086

REFINERY - INSIDE TANKS

Blow Case - Road Tar	5' 6"	22'	4,000
Pitch Tank - Road Tar	6' 0"	22'	4,230

REFINERY - UNDERGROUND TANKS

1	Vapor from Tank 20	5' 9"	16' 0"	3,120
2	Not in use	6' 0"	14' 9"	2,880
3	Heavy Creosote Oil	6' 2"	58' 6"	13,070
4	Creosote Oil	6' 2"	65' 5"	14,616
5	Water from Stills	7' 5"	16' 0"	5,200
6	Creosote Oil	7' 10"	40' 0"	14,420
7	Tie Treating Oil	6' 0"	50' 0"	10,716
8	Fuel Oil	6' 0"	50' 0"	10,716
13	Fuel Oil	5' 5"	20' 0"	4,260
15 *	See Foot Note	8' 0"	26' 1"	10,116
17	Not in use	6' 0"	23' 2"	5,130
18	Not in use	6' 0"	23' 2"	5,130
RCX-8	Not in use	5' 7"	26' 2"	4,930

* Storage 15 is RCX-72 and raised up from ground during high water. The tank is lined up for installation next to Storage 17.

RCX-8 was used for fuel oil to supply the boilers when they were located in refinery building.

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134,000

120,

110,000

10

90,

80,000

70,000

60,000

50,000

40,000

30,000

20,000

10,000

0

1951

35

36

37

38

39

40

41

42

43

44

45

46

47

48

49

50

51

- total tar used
- coke oven tar
- water gas tar
- vertical retort tar

Figure 1

Bued on Dec # 400,500
 entitled "St. Louis Park Plant
 Tar Purchases"

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c) DISTILLATION PROCESSES

The major activity at the refinery was obviously the refining of coal tar. The refining consisted of distillation followed by various blending, purification and separation processes. The types of processing of the distillation products changed over the years presumably due to the market for the various products. The temperatures and time lengths of distillation also fluctuate due to type of tar distilled and desired products.

A typical horizontal batch still (similar to those used at St. Louis Park) is pictured in Figure 2.

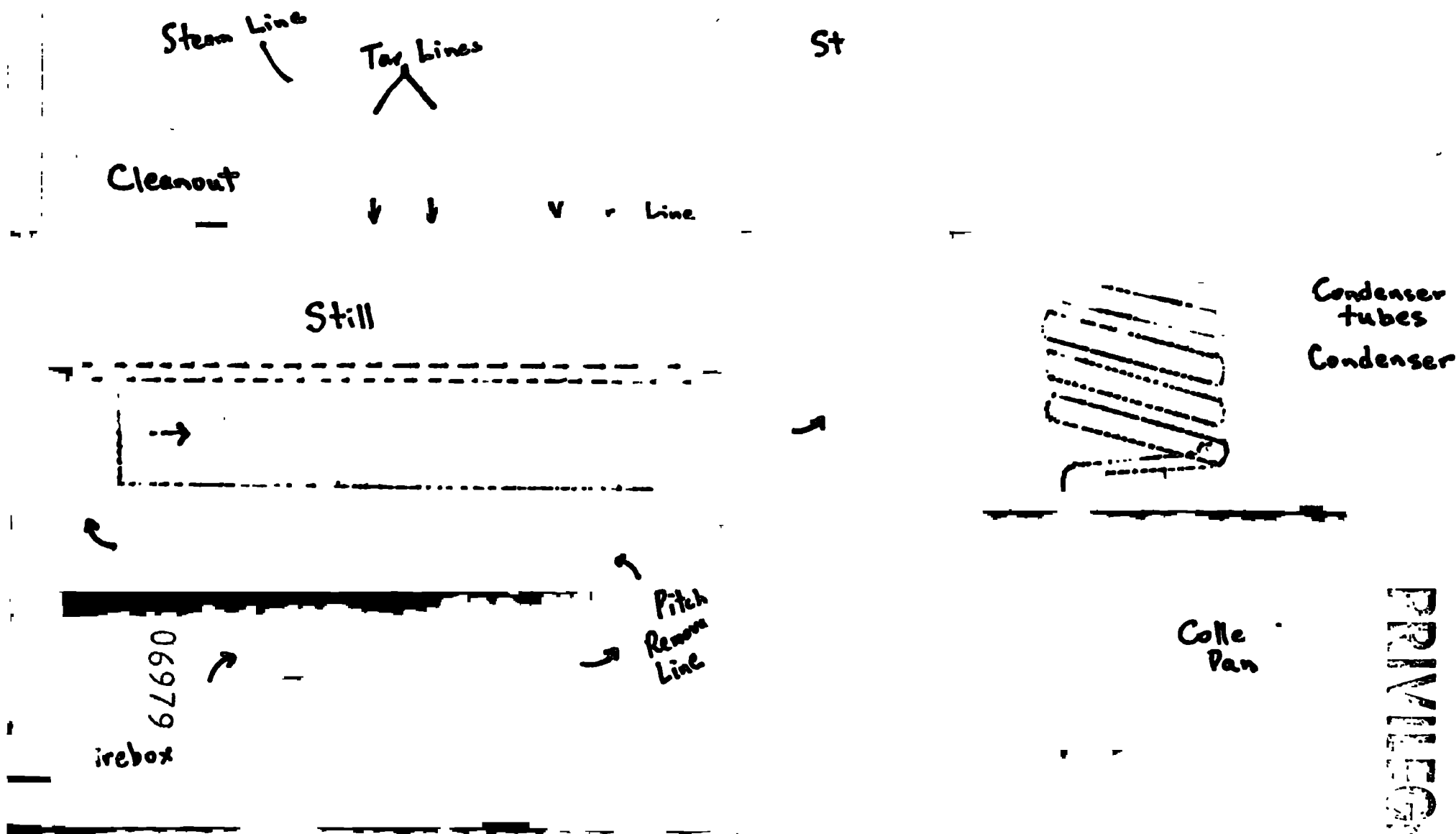
The stills used at the St. Louis Park plant were insulated horizontal cylinders 16 feet long by 7.5 feet in diameter. They were heated by the use of hot gases produced mainly by the burning of fuel oil and natural gas as previously noted. Other fuels used to a much lesser extent were water-gas tar and coal. These were used at times of low availability of the major fuels or in attempts to find lower cost fuels.

The stills were charged with tar by pumping from one of the storage tanks or tar cistern to the stills via one of the tar inlets. The tar was pumped at a rate of about 200 gallons per minute (gpm); therefore it took about 20 minutes for a typical filling. The stills were usually filled to about 4000 gallons although they could hold up to 5200 gallons of tar. At times during the 1920's the typical charges consisted of down to 2500 gallons per still.

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Typical Still - Condenser System

Figure 2.



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When the still is in operation the vapors from the tar leave at the top of the still and enter the vapor line which leads to the condenser. The condenser consists of a tank filled with cool water through which the vapor line makes many passes. The vapor is condensed to a liquid state in the condenser and is then collected in a collection pan. These pans held about 550 gallons of condensate. From the pans the condensate would be pumped to various storage and processing tanks for future use, storage prior to shipping, or further processing.

Further description of the stills and appurtenances is necessary. The hot gases from the fuel combustion are enclosed within brick walls which also support the still. These gases are allowed to surround the bottom half of the still which is confined by brickwork. The hot gases then are discharged to the stack. Other appurtenances to the still include steam lines, pitch removal line, and removable manhold covers (for cleaning purposes).

A typical operating sequence for a still follows:

After a still has been charged and the fire started it typically was about two hours before the first material was vaporized. For the first two to three hours after distillation began the distillate was mainly water. This portion of the tar was called the wet cut. At the sign of oils entering the collection pan this initial condensate was disposed of. At this time, steam was introduced into the still to produce agitation of the contents, thus aiding in the distillation. The second cut,

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or first oil cut, was then collected in the pan. Apparently, at the St. Louis Park plant four cuts of oil were taken from a 4000 gallon charge of tar. It appears from Doc. #303664, a description of procedures for operating the stills prepared in 1957, that the cuts were determined only by volume. Each cut (after the wet cut) was about 550 gallons based on the capacity of the collection pan. This lack of sophistication regarding classification of cuts does not necessarily apply during the earlier years of plant operation. During these years more types of products were produced than in the 50's and 60's; therefore more careful control on separation of the various cuts was probably used.

After the volatile components of the tar (those that are vaporized) are distilled off, the remaining material (about one-half of the tar by volume) is called the pitch. This pitch may be blown out of the still through the pitch removal line by using steam or it may be further heated to produce coke. The coke would then be quenched with a few hundred gallons of water and then be removed from the still by shovel. Pitch may be cooled somewhat in the still but usually was cooled in the pitch bay. From there, in a hardened state, it would be loaded onto boxcars for shipment. Coke, a solid product, would be handled similarly.

d) Products and Further Processing:

As previously indicated, the following cuts may be taken during coal tar distillation:

Wet cut	(until oil is distilled)
Light Oil	(up to 200°C)
Middle Oil	(200°-250°C)
Heavy Oil	(250°-300°C)
Anthracene Oil	(300° - end of distillation)

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The residue left in the still after distillation varies with length of heating and final temperature of the distillation. At St. Louis Park the main control for final temperature was time. To produce coke required about 13-15 hours. Various types of pitches were produced in 8 1/2 to 13 hours. Road tars usually only required about 7 hours. Obviously the time for tar was subjected to heating affected the composition of the anthracene oil (or heavy oil if the anthracene oil was not kept separate.)

The pitches and road tar were sometimes processed further to meet users' specifications. Normally, the pitches could be blended with pitches of different composition to meet the requirements. Road tar could be blended (or cut back) with some of the distillation products, if necessary.

Further processing of the distillation products results in products such as tar acids and bases, naphthalene, and various weights of oils.

The light oil fraction may be redistilled and further purified and washed to give benzene, toluene, and xylenes. The high-boiling residue is then mixed with the middle oil for further processing (Ref. 3 p. 485). Tar acids may be removed prior to redistillation by alkali addition (Ref. 2. p. 90).

The middle oil may be cooled in large shallow pans to crystallize out the naphthalene. The naphthalene is removed by centrifuging the oil; then it is further purified by distillation, washing with sulfuric acid, water, and aqueous

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alkali and distilled again. The oil is washed with aqueous sodium hydroxide to extract the tar acids. From the aqueous solution the tar acids are released by use of steam and carbon dioxide. They are then distilled to form phenol. The oil is then washed with dilute sulfuric acid to extract the tar bases which are further refined to form pyridine. Further processing of the oil may lead to final products of solvent naphtha and coumarone resins.

The heavy oil may be treated the same as the middle oil to produce naphthalene, tar acids (cresols), and tar bases (quinolines).

The anthracene oil (if collected separately) may be allowed to cool and crystallize (one to two weeks). The solids are then dried and ground. Washing with solvent naphtha removes much of the solid phenanthrene. Washing with pyridine removes much of the carbazole. Anthracene is then produced from the residue by sublimation. (Ref.3 p. 485) Anthracene ^{was} ~~used to be~~ an important intermediate in dye manufacturing but has not been important for 30-40 years, due to dye intermediates being synthesized from benzene. Therefore most anthracene oil was not collected after the 1940's but was left as creosote oil along with the heavy and middle oils.

The St. Louis Park plant did perform some of the operations described in the preceding four paragraphs. Up until 1930 (See Doc. #103466, memo dated May 7, 1930.) The light oil was processed to produce tar acids, pyrabasic, and residual oils.

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Light oils and heavy oils were sold as distilled in various cases. Middle oil was processed to remove naphthalene, tar acids, and tar bases in the early years of plant operation. Naphthalene was probably also extracted from the heavy oil. We have no records, however, of further processing or even the collection of a separate fraction of anthracene oil

The plant supplied the creosoting plant with creosote during most of its existence. That demand for creosote and outside demand probably led to greater amounts of all of the distillate fractions to be used as creosote oil than at a typical coal tar refinery.

The refinery weekly reports present the amounts of products produced in the distillation process. It does not appear that they refer to further processing of the distillation products done in the by products plant. Information regarding by products production may be obtained if we have all of the weekly reports and inventories and the monthly and yearly inventories for the plant. Lacking those we can, however, make some estimates of levels of production of distillation products. Figure 3 presents data on these production levels for the years 1934-1971. This figure is only a crude indication due to the extremely small number of weekly reports used to generate it. However, it is safe to say that it does show production trends. Similarly Figure 4 shows the trends of production of the various types of residues.

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From Figure 3 it is obvious that throughout the plant's lifetime, creosote oil was the major distilled product. During the early years of the plant (until 1940) a significant amount of light oil was kept separate from the creosote oil for further processing in the by-products plant. In the early 50's much light oil was recovered and ^upresumably processed further. This was apparently due to a low demand for the creosote oil and/or treated wood products from the creosoting plant. After about 1960, light oil was apparently not kept separately from the creosote oil. A June 20, 1963 memo (doc. #100396) refers to the removal of the byproducts building. In the 1960's some heavy oil was collected. It is my opinion that this oil was used to cut back the pitches and/or road tar produced at the plant to meet customers' specifications. ✓

From Figure 4 it is apparent that the emphasis on the residual products changed over the years. Coke was a major product of the refinery from its opening until about 1955. Various pitches (fuel, soft and roofing pitch) were produced throughout the plant's life. Road tars were very important in the 1950's and were also a significant product up until the early 1960's. Heavy electrode binder pitch, which was blended with other pitch (normally obtained from Gary, Indiana) to produce complete anode pitch, was the major residual product from the mid-50's until the late 60's. During the late-60's a pitch called "hard pitch" was produced; this may have been the same as

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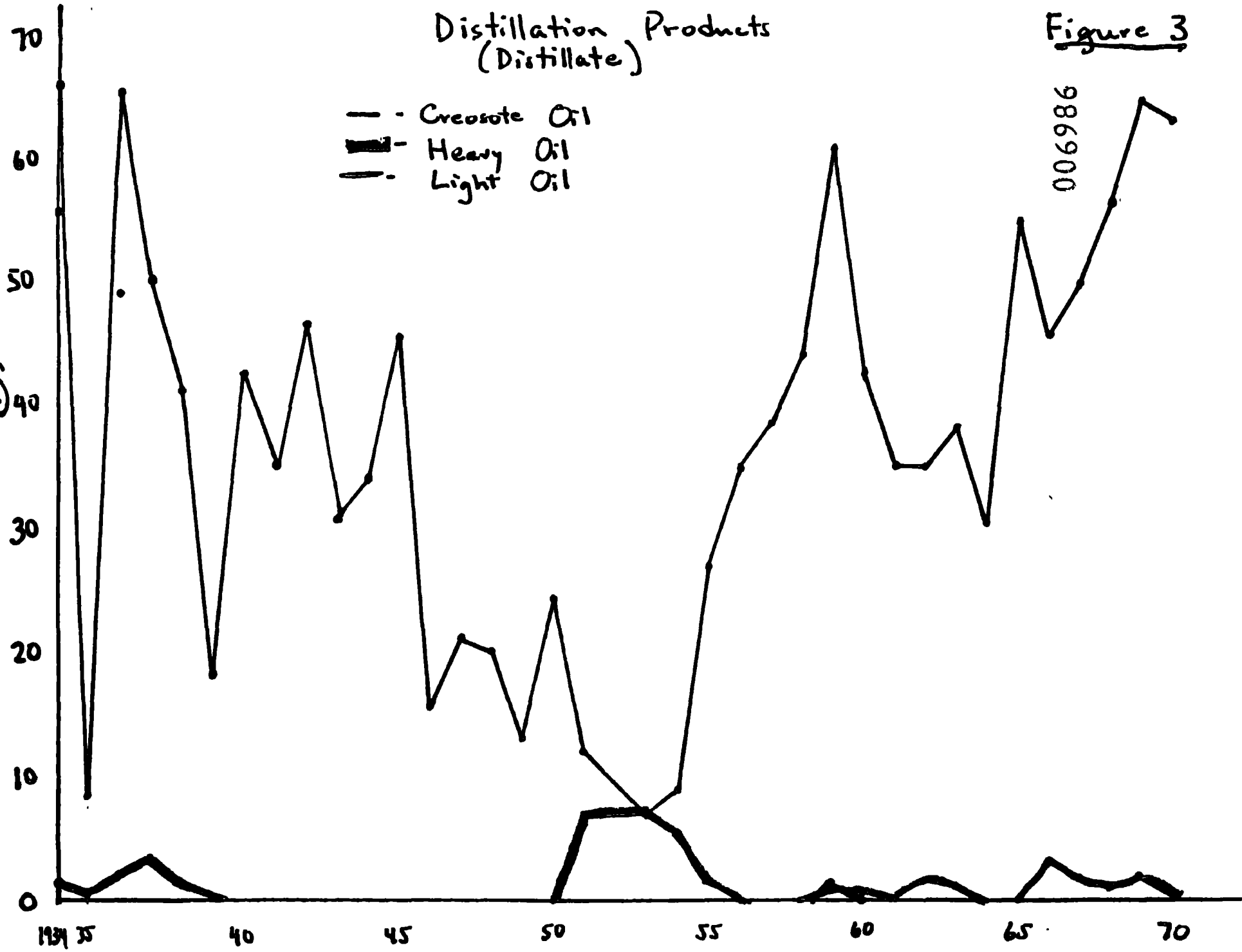
Distillation Products (Distillate)

Figure 3

- - Creosote Oil
- - Heavy Oil
- - Light Oil

Production
(1000 gal/yr)

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Distillation Products (Residues)

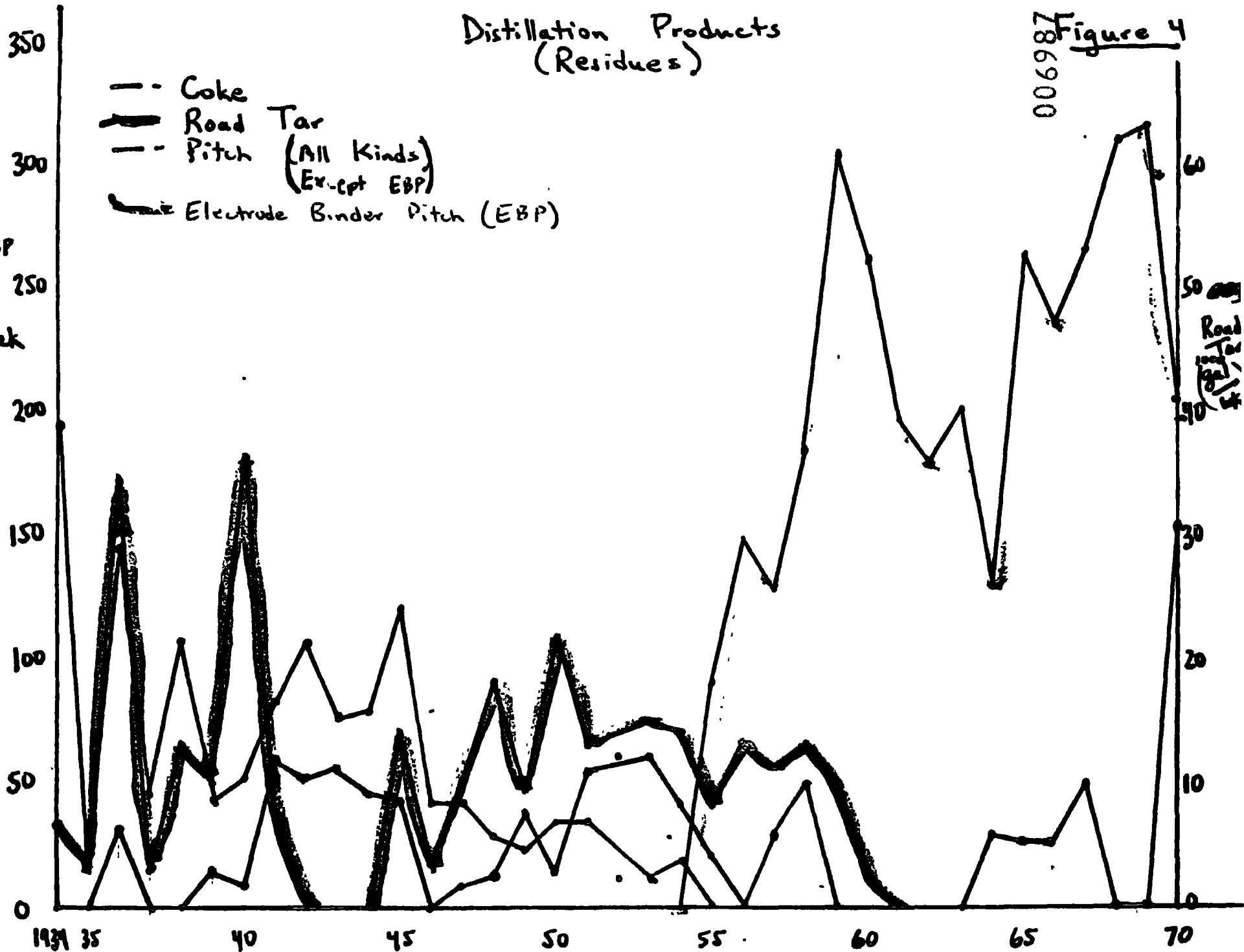
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Figure 4

- - Coke
- Road Tar
- - Pitch (All Kinds)
(Except EBP)
- Electrode Binder Pitch (EBP)

Coke,
Pitch, EBP

ons/week

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the heavy electrode binder pitch. The complete anode pitch was a blend of about 2 1/2 parts heavy electrode binder pitch to one part Gary pitch (or similar pitch). This product was used by the aluminum industry in the production of aluminum.

Since the products produced at the refinery were all merely portions of the original coal tar (that is to say that few, if any, chemical reactions took place during any of the processing steps to form different compounds), what we know about the composition of the coal tar is most important in predicting what types of pollutants may be found in the ground water from this site.

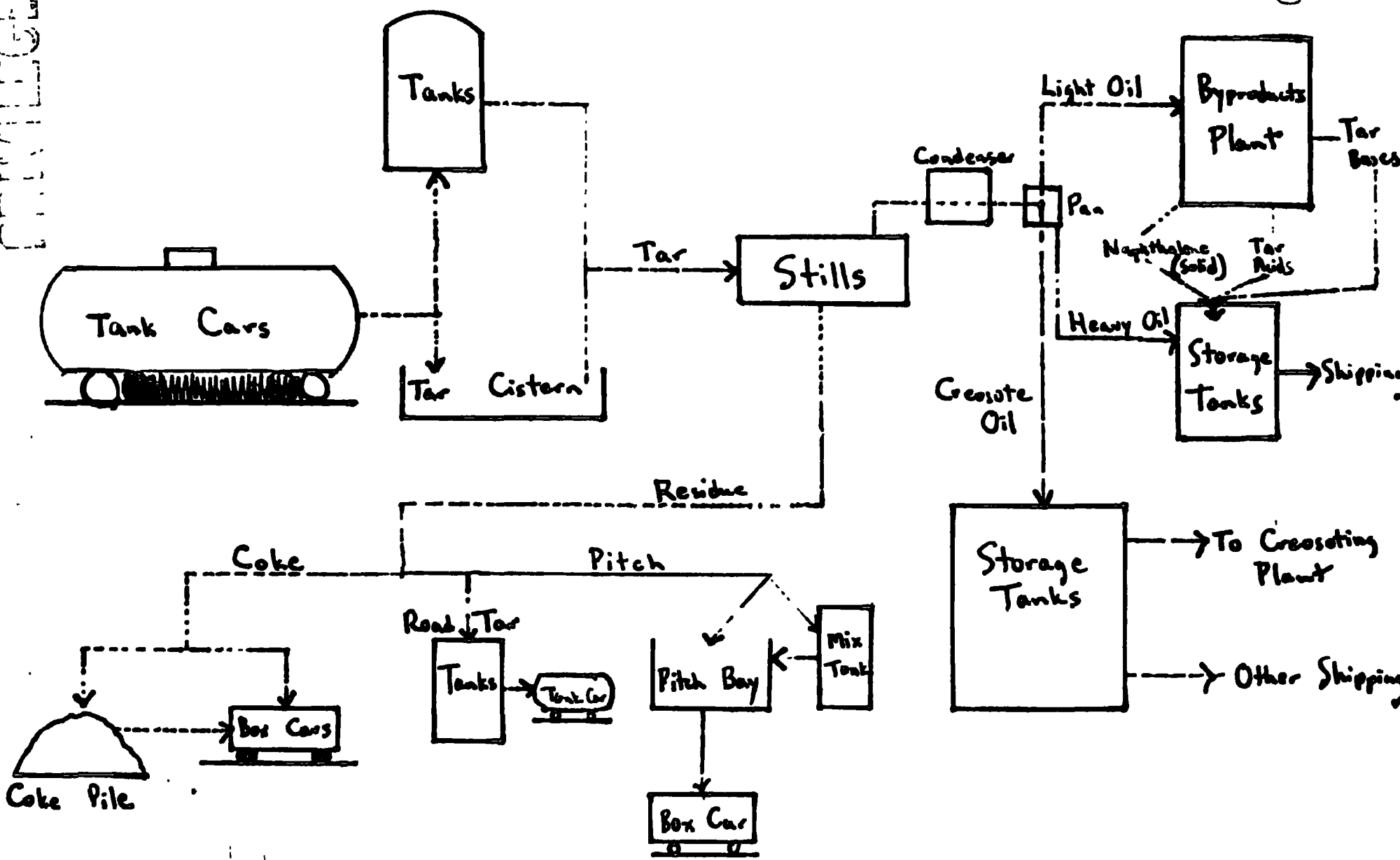
Figure 5 presents a simplified flow chart of raw materials and products for the refinery. From this figure it is obvious that numerous opportunities for leaks or spills of materials existed. Several documents we obtained from the Company indicate that the piping (underground) and the storage tanks were in extremely poor condition. In the late 1960's the decision was made (see document #220562 a work order dated June 13, 1968) to eliminate all underground piping and tanks. Apparently this was necessitated by excessive losses of materials although we cannot even estimate the amount of losses from the information we have.

Other plant systems which were important in the tar processing were the plant's water system and the boilers.

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Refine Material Flow Chart

Figure 5

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An inspection report by J.A. Lauck dated July 22, 1954, (Document #305156) provides much information regarding the operation and condition of both of these systems.

The water used at the plant was obtained from a 1000 foot deep well according to a September 21, 1945, memo (Document #215481). The water was pumped from the well to an open pond from which it was pumped for various uses. This pond was described in several memos and letters between 1945 and 1954 - (See Documents #215481, #215983, and #305156.) The pond was said to be dirty and normally covered with an oil slick. Due to the pond's contamination buildup of scale and oil in the boilers and condensers was a problem at the plant.

The plant used two boilers for steam production. The boilers were located in the boiler house and were rated at 300 horsepower and 150 horsepower. It appears that at least the larger boiler was constructed in 1940. Before this date, the smaller boiler may have been the main boiler. However, Document #215481 (memo dated September 21, 1945) indicates that the 300-horsepower boiler was necessary to be operated to provide enough steam for both the refinery and creosoting plant operations. At this time, the 150-horsepower boiler was used mainly to burn wood scraps from the creosoting plant operations. Apparently both boilers were used until plant closure as the "Republic Creosoting Company, Plant Operation and Property Description" written in 1971 (Doc. #401877) mentions both boilers (although the capacities are presented as 110 and 310

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horsepower). No indication was found in the documents that these boilers were replaced after 1940.

As indicated previously, the boilers were used to provide steam for plant operations. In the refinery, steam was used to agitate still contents during distillation, to heat raw materials and products in the storage facilities to facilitate pumping and to prevent solidification of products, to blow pitch from the stills, and to heat the buildings. The steam was produced by the heating in the boilers of feed water from the water supply pond. The water was heated in the boiler tubes which were in general in poor repair according to several insurance inspection reports. Leaks and explosions were possible due to this condition; however, we have no indication from the documents that these things occurred. Even if they did, little contamination of the ground would be expected from such an occurrence.

The condition of the boiler tubes is presented here mainly as an indication of the general condition of plant equipment. While several insurance reports suggest that these tubes be repaired or replaced, it is apparent that these suggestions were largely ignored by the Company. The condition of the tubes did cost the plant money as heat transfer is impaired by dirty tubes and heat is lost through steam leaks. This is important because it shows that the plant was allowed to deteriorate in several areas, even though it might have made

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sense (money) to perform some of the necessary maintenance activities. The Company similarly lost steam and products through leaky tanks and underground piping for many years before spending the money necessary to eliminate this problem.

The plant was subject to various incidents which may have caused loss of materials to the ground on the site over the plant life. Our records are more complete during the last ten years of plant operation, but we are aware of some previous cases. The following table lists some of the fires, explosions, overflows and other similar events which may have contributed to ground contamination. Most of the reports we have did not include the amount of material lost so we cannot determine a total amount lost due to these types of events. I have each of the documents listed on the chart in a separate file.

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FIRES, EXPLOSIONS, SPILLS, ETC.

Date of Document	Date of Event	Type of Event	Material Lost	Amount Lost	Where?	Cleanup?
6/30/38	?	Explosion & Fire- Stills 1/4	Tar?	?	Refinery	?
1/4/44	12/27/43	Fire- Kettle	Fuel- Pitch	?	Refinery	?
10/7/58	contin- ous	Leaks in Steam lines	Steam	?	Storage tank & Un- loading areas	----
4/17/63	4/15/63	Explosion	Pitch	a lot	Pitch Blend Tank	Planned
12/24/64	12/18/64	Fire	?	?	Refinery	Inside
6/2/65	6/1/65	Fire	?	?	Tank #20	?
9/21/66	9/14/66	Fire	?	?	Tank #20	?
7/13/67	10/3/66	Explosion	Tar	0	Tank #20	NA
10/26/66	10/13/66	Fire	--	?	Tank #19	?
2/1/67	1/25/67	overflow Fire	Tar	Little	Still #16	-----
5/2/67	4/25/67	Explosion- Fire	Tar	9300 gal	Still #11 Refinery- outside	?
8/22/67	8/12/67	Fire	Tar	0	Stills #13, 14, & 15	NA
3/21/68	?	Boilover	Pitch	?	Still #14	?
4/11/68	3/29/68	Overflow	Tar	?	Still #15	?
2/17/69	?	Leak	Tar	600 gal.	Still #13	Yes
5/7/69	5/1/69	Fire	?	?	Still #16	?
5/27/69	5/24/69	Fire	----	0	Still #16	NA
10/3/69	9/15/69	Rupture	?	?	Still #16	?
1/27/71	1/27/71	Fire	?	Slight	Still #15	?
5/21/71	?	Fire	----	0	Tank #20	NA

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One other area of interest is the replacement and repair of equipment at the refinery, specifically the stills. From various documents, the history of the refinery in this regard may be pieced together. While repairs to the stills were quite common (such as replacing insulation damaged due to saturation with spilled tar), replacements or installing new still bottoms were significant events. These events were basically necessitated by the weakening or cracking of still bottoms due to age. In other words, the stills were run until the bottoms wore out.

The original refinery apparently contained two batteries each containing four stills. In 1929 a third battery of four stills was added. A fourth battery followed in 1936. A single still was also added about this time. The stills were numbered one through 17 in the order just presented.

Stills #1-4 were replaced in 1939. #5-7 were replaced in 1941. A new bottom was installed on #17 in 1943. #15 and 16 were replaced in 1944 and #13 and 14 were replaced in 1946. New bottoms were installed on #5-8 in 1945. New bottoms were installed on #9-12 in 1947. #1-4 probably were not used past 1945. As of 1954 #1-8 were no longer used. #11 and 12 were replaced in 1956 and #9 and 10 were replaced in 1958. New bottoms were installed on #13-16 in 1958. These four stills were replaced with five-tube conversion stills in 1961. These fire-tube stills apparently operated with the hot gases (from

e) Wastewater Sources

The main source of wastewater from the refinery was the wet cut which was previously defined. This wastewater stream was contaminated with many of the more volatile components of the coal tar. During the late 1950's the refinery weekly reports quantified the wet cut for the first time. It appears, from these reports, that about 5 per cent of the coal tar refined was collected as wet cut and disposed of. If this percentage was typical over the plant's life, this wastewater source varied from 2000 gallons per week (in the early 1930's and late 1930's) to 6500 gallons per week (in the mid 1940's and in the 1960's). The average flow over the plant's life was about 4500 gallons per week of wet cut.

Other apparent wastewater sources included condenser cooling water, boiler water blowdown, still cleaning water, coke quench water, water from stored raw materials, laboratory wastes, and sanitary wastewater. Contaminated surface runoff from storage tank areas and the piping trenches might also have entered the plant's wastewater system.

The condenser water and boiler blowdown were essentially uncontaminated streams. However, if steam coils were leaky or if the condenser were leaky, these streams could have become slightly contaminated with tar or other materials. Also, the source of the plant's raw water was the open pond previously described which was said to be somewhat contaminated. The boiler blowdown and condenser water could be reused; however,

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the amount of pollutants discharged to the wastewater system would not be significantly affected if they were or not. From the information we have, it is unclear how the stills were cleaned and if wastewater was generated.

Coke quench water was used in the stills to cool the coke rapidly so that it could be removed from the stills. This operation used up to 300 gallons of water per still which was discharged to the wastewater system. This could have contributed up to 6000 gallons of wastewater per week in the 1930's and until about 1945. For the next ten years this figure might have been about 2000 gallons per week. After 1955 coking was typically done at the refinery.

If water was in the raw tar shipped to the plant it may have separated in the storage facilities. Common practice at coal tar refineries was to drain water off the tar if it did separate noticeably. This source of wastewater may not have existed at St. Louis Park but if it did it would be expected to be contaminated with many tar components.

Other wastewater sources were laboratory wastes and sanitary wastes. These streams apparently went to the wastewater system as we know that sanitary sewer was not available at the plant site. (Several documents of the late 1960's and early 1970's discuss the possibility of having sanitary sewer installed to service the plant.) These streams are relatively small and insignificant.

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To summarize, contaminated wastewater probably amounted to 6500 to 10,000 gallons per week from the refinery. Other small flows or relatively uncontaminated flows might push the figures slightly higher.

CREOSOTING PLANT

a) Raw Materials and Production

The raw materials used in wood treating are the wood and the treating material. At the St. Louis Park plant the wood used was a combination of hardwood and soft wood species. The major products of the plant were treated cross and switch ties for the railroads. However, Wheeler Bridge Company was also a significant customer. We have weekly reports of treating operations between 1935 and 1957. We do know that the treating plant did operate until the refinery closed from refinery reports. The treating plant reports after 1942 do not ^{indicate the} ~~indicate that~~ customers, but we do know that the railroads were customers for the life of the plant. Also, Wheeler Bridge was operating in the 1960's; therefore we can assume that it was also a customer until the plant closed.

The major treating material used at the plant was creosote oil. The use of creosote oil as a wood preservative was first shown to be effective in the 1830's and still in 1977 was used almost exclusively for the treatment of railroad ties and bridge timbers. (Ref. 7, p. 21) The creosote oil imparts a dark oily appearance to treated wood and the surface is not suitable for painting. Therefore other "cleaner" types

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of preservatives are now used for products when appearance is important (such as windows, doors, deck lumber, landscape lumber, etc.). This was not a real consideration for the type of treating done at this plant. Also, being tied in with the refinery it would not have been economically advantageous for them to abandon creosote treatment.

Other preservatives used at St. Louis Park included zinc chloride solution (until the mid 1940's), petroleum oil (probably similar to #2 fuel oil) which was mixed with the creosote oil in various proportions (usually 40-50% petroleum oil), and pentachlorophenol which apparently was used in a 2% solution with with some type of light petroleum oil or organic solvent after 1959. Since we have no weekly reports after 1957, it is unclear to what extent pentachlorophenol was used at the plant.

Production levels are quite consistent over the periods for which we have reports. The use of creosote oil is related to amounts produced at the refinery; however in the 1960's much of the creosote oil produced was shipped elsewhere. During the early years of the treating plant creosote oil usage was lower because of the use of zinc chloride. During the early and mid 1940's production levels were somewhat elevated (averages of about 50,000 cubic feet of wood treated per week versus 35,000 cubic feet per week).

b) Wood Treating Processes

Prior to treating, the wood is debarked, sized, incised,

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and dried. Debarking was apparently not done at the St. Louis Park plant but the other processes were. Sizing was done in the so-called adzing and boring mill. Incising, which consisted of making small holes in the wood to facilitate preservative uptake, also was done at this mill (at least after 1961, the year an incisor was purchased according to Doc. #220410). Drying of the wood was accomplished by allowing the wood to stand in the open for 12 to 14 months prior to treating, according to Doc. #401877 (Plant Operation and Property Description prepared in 1971). We have little information to support the belief that this was the sole method of wood drying throughout the plant's lifetime.

Other methods of drying include tunnel drying, kiln drying, and various types performed in the treating vessels. Tunnel drying consists of slowly moving the wood through a heated tunnel for up to about two weeks. Kiln drying is a batch drying process which can be controlled as to temperature and retention time in the kiln depending on wood species and moisture content. (See Ref. 6, p.4-3).

Drying processes which are performed in the treating vessels include open and closed steam conditioning, vapor drying, and Boultonizing. Open steam conditioning consists of adding steam directly to the closed vessel containing the wood. Closed steam conditioning consists of heating water in the vessel with the wood by passing steam through coils at the vessel's bottom thus vaporizing some of the water. In

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both open and closed steaming, water from the wood is evaporated thus drying the wood. The steam released from the vessel in both cases is condensed. In the open system this condensate which includes preservative from the vessel is wasted. In the closed system this condensate is returned to a reservoir to be reused similarly. A small amount of this water is wasted to keep levels of preservative and dissolved solids at a satisfactory point. The steam used in the coils is also condensed and reused in the boiler. The boiler, by the way, is the same 300 horsepower boiler used for refinery operations.

Vapor drying utilizes high boiling-point organic liquids to remove the wood moisture. These organics are vaporized and introduced into the closed vessel. They condense on the wood surface and the heat of condensation is transferred to the wood thus vaporizing the water from the wood. The condensed organics and water are separated with the organics reused and the water wasted.

Boultonizing is a drying process which is combined directly with the treating process. The wood is immersed in preservative heated to 82°C to 99°C. The vessel is closed and a vacuum drawn to lower the water's boiling point. The vapors from this product consist of water and preservative component and are condensed and wasted.

From the information we have it is not possible to make a definite determination of which drying methods other than air drying, if any, were used. However, we do know that in

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PRELIMINARY

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1954, a 47,000 gallon tank at the creosoting plant was used to hold water. (See Doc. #305156, an inspection report dated July 28, 1954.) It seems that this could have been the reservoir used in a closed steam conditioning system. We do know that steam coils were in place in the treating vessels, another necessity for a closed system. A typical closed steam conditioning treating system is presented in Figure 6.

The treating vessels consist of tanks with hinged doors that provide air-tight seals. The tanks are cylindrical and are 4 to 10 feet in diameter and up to 175 feet long. The wood to be treated is loaded on metal trams by the use of lift trucks. The trams are wheeled into the treating vessel on steel tracks. The wood is removed after treating in a similar fashion and stacked in the yard. Document #401878 (1971 report) indicates that the railroad ties were immediately loaded onto railroad cars for transport.

The St. Louis Park plant had 3 treating vessels which were about 150 feet long. While it is possible that the railroad ties, after treatment, were sometimes immediately removed from the plant area, we can see from the aerial photographs that much of the entire plant ^{site} was used for treated wood storage.

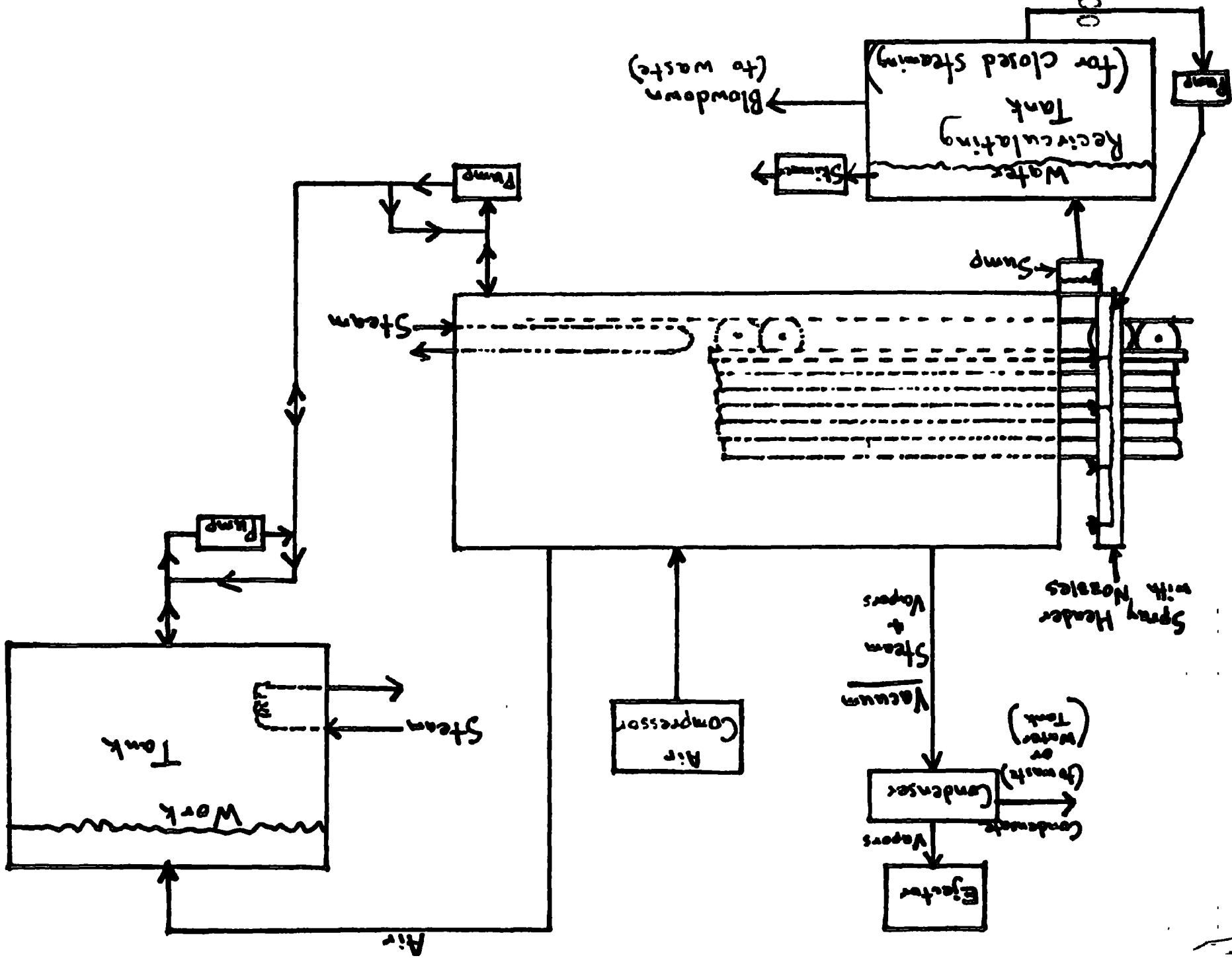
Pressure treating, which was the preservative application method used at St. Louis Park, is either done by the full cell process or an empty cell ^{process. The terms} refer to whether the wood's cells are filled with preservative or the cell walls are merely coated with preservative.

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Figure 6

Probable Treating System



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The full cell process is initiated by placing the wood in a vessel and applying a vacuum. Then the vessel is filled with the preservative without admitting air. Pressure and heat are then applied to force the preservative into the evacuated cells. A final vacuum may be applied to remove excess preservative from the wood. This process results in maximum protection of the wood and is typically used on wood to be used in marine applications.

The empty cell processes are the Lowry and Ruepping processes. The Lowry process consists of placing the wood in the vessel, adding the preservative, applying pressure to force the preservative into the wood, and applying vacuum to remove excess preservative thereby only leaving the wood cell walls coated. The Ruepping process is the same as the Lowry process except that the vessel is air pressurized prior to addition of the preservative. This process allows less preservative to enter the wood than the Lowry process. Both of these processes, however, are capable of deep penetration of preservative (Ref. 6, p. 4-13).

The Ruepping process generally has a preservative tank to maintain pressure in the treating vessel during the preservative addition. This tank receives the air from the treating vessel as the preservative is sent to the vessel.

The St. Louis Park plant normally used the Ruepping process for wood treating. This process produced treated wood satisfactory

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for uses such as railroad ties and it was the process which used the least amount of preservative per volume of wood treated. Three of the four work tanks at the treating plant could be used as Ruepping tanks according to December 28, 1961 memo (Doc. #200101) which also states that " . . . the majority of our treating requirements are processed by the Ruepping method." This memo also states that one of the work tanks can only be used for the Lowry or full cell processes.

In 1962 two new work tanks were installed to provide greater flexibility. Around 1960, the Company began using a creosote-pentachlorophenol treating solution which necessitated one work tank be kept specifically for this purpose (Doc.#200101).

Also, the Company wanted to treat bridge timbers for the Soo Line Railroad with Creo-Arban. This also required separate work tanks. (Creo-Arban must be a brand name as I have not been able to determine what it is, although it probably is mainly creosote oil with some other preservative or fire retardant in small amounts.)

c) Pollutant Sources

Wastewater sources for the wood treating process include steam conditioning condensate, condensate due to the vacuum during treating process, washwater, surface runoff from storage areas, drips from just-treated wood and ground around the treating vessels. Also boiler water contaminated with preservative by leaky steam coils in treating vessels or storage tanks.

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These streams can obviously be contaminated by preservatives (creosote oil, petroleum oil, zinc chloride and pentachlorophenol) and also by wood sugars dissolved in the steam and water. Typical amounts of wastewater for a closed-steam conditioning, Ruepping process wood treating plant would be about 5000 gallons per day (Ref. 6). This figure would not include surface runoff, which probably was not collected at St. Louis Park. This figure could range from 1000 to 12,500 gallons per day.

Ground contamination occurred from leaky pipes and tanks, storage of treated wood (outside of heating area) and spills and leaks from the treating area. A sump in the treating building did collect some spills but others must have soaked into the ground.

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3) WASTEWATER TREATMENT

Previously I estimated total wastewater from the site (excluding stormwater runoff) at about 5000 gallons per day from the creosoting plant and about 10,000 gallons per week from the refinery. This would result in a weekly flow of about 35,000 gallons of wastewater. This figure varied somewhat as previously described due to production volumes and processing changes, but should represent a typical figure for most of the plant's life time.

The earliest information we have regarding wastewater disposal is contained in a May 1938 inspection report (Doc. # 219256 - 219260) by the state health department. This report indicates that the wastewater was discharged to a ditch running across the plant site to a peat bog immediately south of the site. This bog had an area of about 4 acres and was divided by Highway 12 which was constructed through it in 1936. The report indicates that the ditch was running at between 150 and 200 gallons per minute (mostly surface runoff) and the water in it contained floating emulsified oil and settleable tar. A sample of this ditch water was analysed; it contained 4400 parts per million (ppm) total solids, 50 ppm phenol and 1900 ppm total volatile matter. The report also described the swamp as having an oil-covered surface and vegetation therein as being coated with tar. Apparently in 1938 no wastewater treatment facilities were in operation.

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UNCLASSIFIED

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A May 1, 1940 Co. memo deals with the removal of phenol from sodium sulphate solution and the wet cut prior to disposal in the drainage system. This memo states that a tank is to be installed in which these wastes were to be washed with vertical retort neutral oils to remove phenols. The phenol would be transferred from the wastes to this oil upon agitation. We do not know if this was ever done at the plant.

According to a memo entitled "Plant Efficiencies, Economics, and Maintenance" dated July 18, 1962 (Doc. #100418), in 1941 the ditch was replaced with 8 inch tile. Also, plans dated July 20, 1944 show this tile line which leads to a structure identified as the retaining basin. From other documents we know that this basin was a 10' X 16' X 50' basin made of treated wood. A pump was used to send the water from this basin to another 8 inch tile line which led to the above-noted swamp. We do not know how well the basin was maintained, whether or not oil was removed from the basin, and, if so, where the oil was disposed of.

In 1968 it appears that for some reason the Company became aware of the need to improve its wastewater disposal facilities. A May 14, 1968 internal memo by C.F. Leshner (Doc. # 208008) refers to the condition of the settling basin. From this memo it seems that little maintenance was ever performed on the basin. Leshner indicates that the basin contained a high level of solids (possibly from boiler blowdown water) and the effluent's

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"visual appearance and quantity of oil going out of the plant was terrible." If the solids were due to boiler blowdown it seems possible to me that the basin had not been cleaned since its construction in the early 1940's. This memo, among others, also confirms that removal of water from tar storage was a source of wastewater from the plant.

A work order dated June 4, 1968, provided about \$4000 for upgrading the wastewater disposal system. This work was to include cleaning the basin, hauling the residue removed from the basin (disposal site unknown), cleaning the trench system, installing a tank so that boiler blowdown water by passes the basin, and installing a straw filter at the plant discharge (presemably at the property line).

Around 1970 Reilly investigated the possibility of discharging the wastewater to a sanitary sewer. Plans dated December 22, 1970 entitled "Plant Sewer to City Sewer" were prepared which included a new oil separator system as well as the sanitary sewer connection. These plans were never carried out.

One other document relating to wastewater disposal is of interest. This is an internal memo dated March 23, 1971 from R.J. Hennessey to Mr. P.C. Reilly entitled "Water Pollution - U.S. Army waste Materials Permit. This document (#303233) provides descriptions of each of Reilly's plants' methods of wastewater disposal. Basically the other plants had similar disposal systems as at St. Louis Park. However, some did discharge to sanitary sewers in 1971.

007008

APPENDIX A

LIST OF REFERENCES

1. Chemistry of Coal Utilization
2. Asphalt and Allied Substances (Volume II)
Herbert Abraham, Sixth Edition @ 1961.
3. Chemistry of Organic Compounds - Carl R. Noller,
3rd Edition @ 1965.
4. The Chemical Processes Industries - R. Norris Shreve,
2nd Edition @ 1956
5. EPA Development Document - Plywood, Hardboard and Wood
Preserving - April 1974
6. Wood Treating Industry - Multimedia Emission Inventory (Draft)
June 1980 (Prepared for EPA by Acurex Corp.)
7. Wood Preservatives - Treatment Processes and Product
Applications - Paper by Robert D. Arsonault (Koppers Co.)
8. Wood Preserving Effluents and their Treatment - Paper
by Neil G. Richardson (Domtar Limited)

APPENDIX B

Structures of Compounds found in Significant Amounts in Coal Tar:

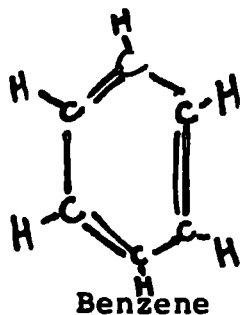
The following table taken from "Chemistry of Organic Compounds" by Carl R. Noller (3rd Edition) presents the major components of coal tar:

COMPOUND	%	COMPOUND	%
Benzene	0.1	Phenanthrene	4.0
Toluene	0.2	Anthracene	1.1
Xylenes	1.0	Carbazole	1.1
Naphthalene	10.9	Crude tar bases	2.0
α - and β -Methylnaphthalenes	2.5	(pyridine, 0.1)	
Dimethylnaphthalenes	3.4	Crude tar acids	2.5
Acenaphthene	1.4	(phenol, 0.7, cresols,	
Fluorene	1.6	1.1, xylenols, 0.2)	

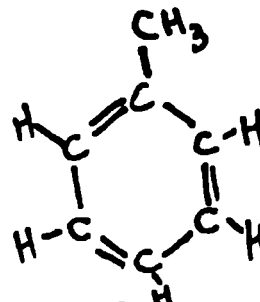
The following are the structures of these major components (and others). If further information about properties and production processes of these components is desired, the above-referenced book may be helpful.

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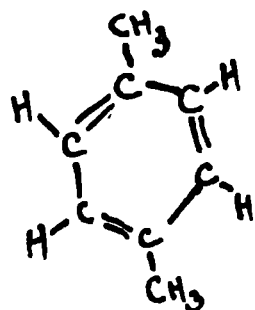
LIGHT OIL FRACTION



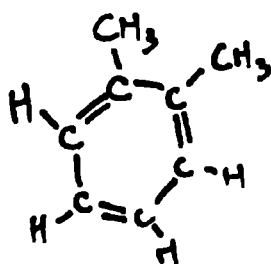
Benzene



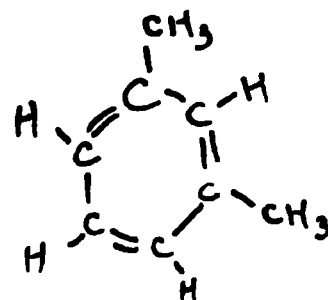
Toluene



p - Xylene



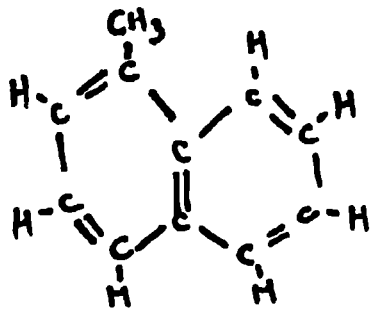
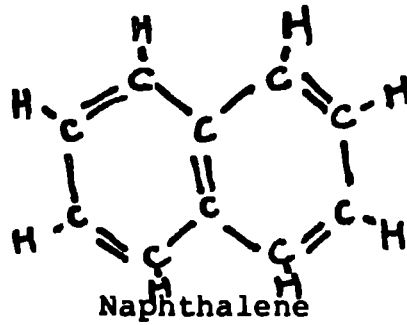
o - Xylene



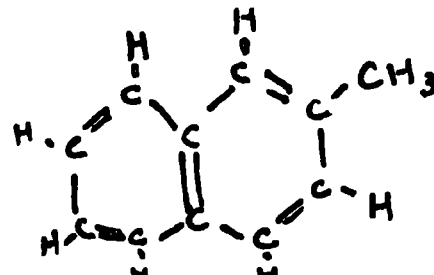
m - Xylene

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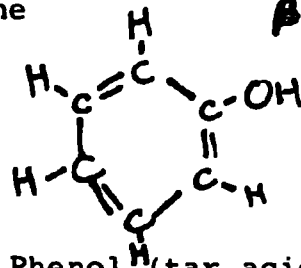
MIDDLE OIL FRACTIONS



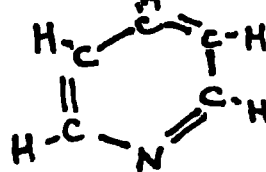
α - Methylnaphthalene



β - Methylnaphthalene



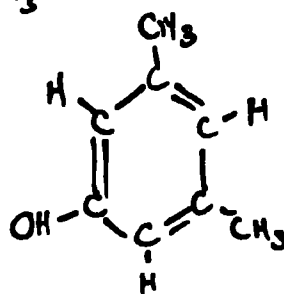
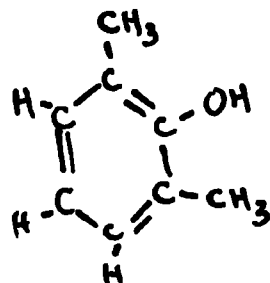
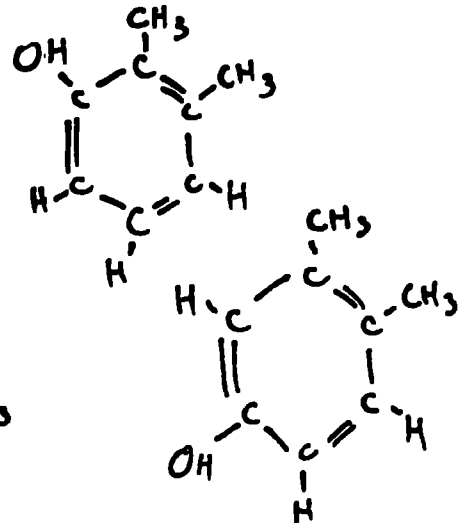
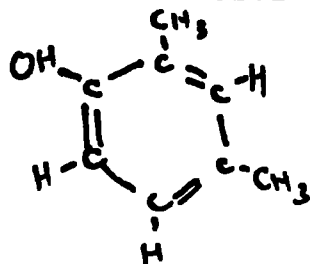
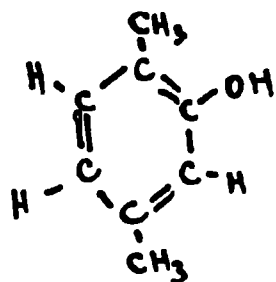
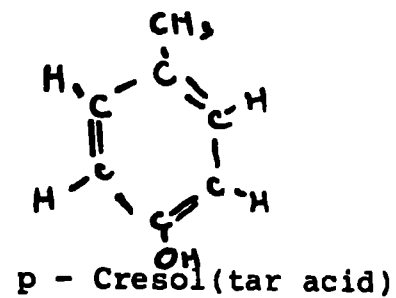
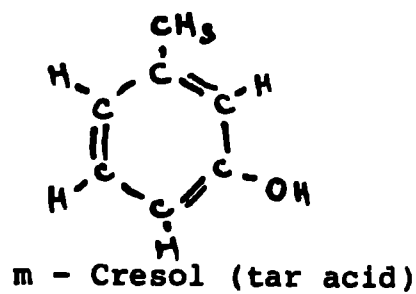
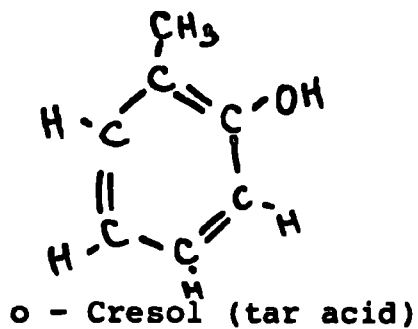
Phenol (tar acid)



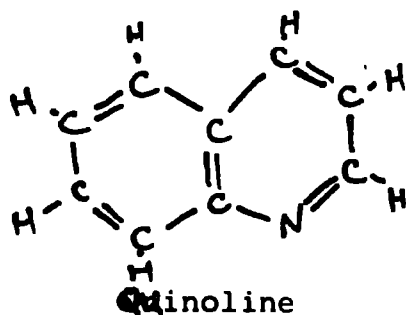
Pyridine (tar base)

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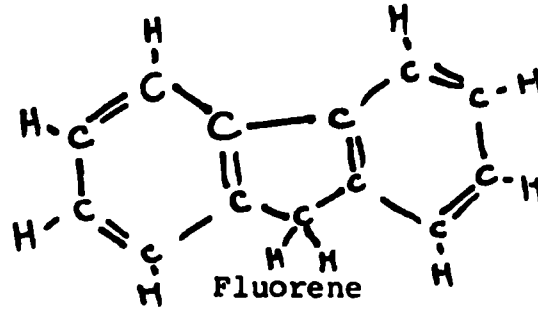
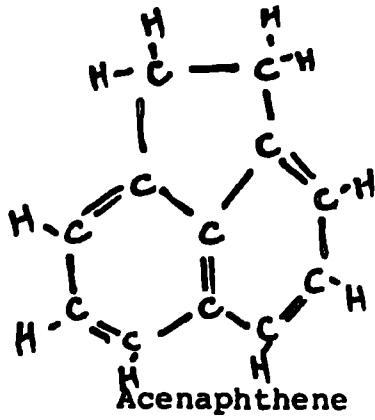


Xylenols (tar acids)

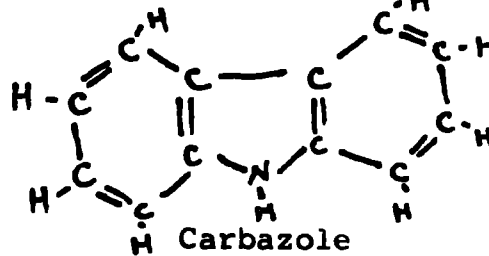
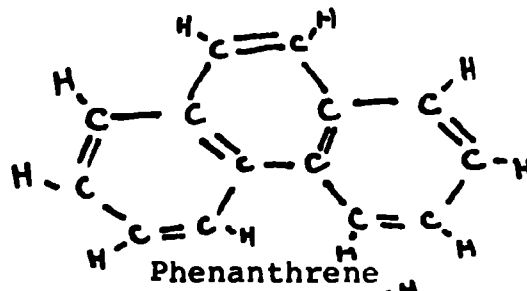
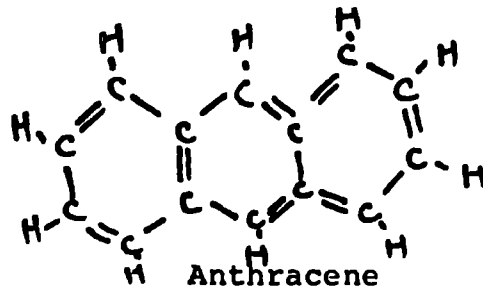


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HEAVY OIL FRACTION



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